

PATENT SPECIFICATION

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(54) TEXTILE PROCESS

(71) We, I.W.S. NOMINEE COMPANY LIMITED, a British Company, of Wool House, Carlton Gardens, London, S.W.1, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for rendering keratinous or other protein material resistant to shrinkage.

Many shrink-resist processes are known which require the application of resins to keratinous fibres and, generally speaking, although the majority of the resins give adequate shrink-resist effects the soil-release properties of the treated fibres tend to be impaired. This is unfortunate because keratinous fibres, and especially wool fibres, have an excellent natural ability to release soil during washing and dry-cleaning, and to resist redeposition of suspended soil in the washing and dry-cleaning liquors.

We have now found a process which will restore the soil-release properties of keratinous and other protein fibres or filaments to substantially their original state without having any deleterious effect on the resin shrink-resist treatment. Furthermore in the case of some resins the process increases still further the shrink-resist effect exhibited by the resin.

The process according to the present invention comprises treating the protein fibres or filaments within a resin capable of imparting shrink-resistance thereto and with a soil-release agent comprising a condensate of terephthalic acid and an alkylene oxide.

The term "protein material" includes all forms of protein fibres or filaments, e.g. fleeces, tops, card sliver, noils, yarns, threads, pile fabrics, non-woven fabrics, woven fabrics, knitted goods and garments. The material to be treated may consist wholly of keratinous fibres or of blends of these fibres with synthetic fibrous of filamentary material such as fibres or filaments of polyamides, polyesters

or poly-(acrylonitrile); or with cellulosic or regenerated cellulosic material. Generally, however, the material should contain at least 30% keratinous material and preferably consists 100% of keratinous material.

The process is particularly useful in connection with the shrink-proofing of wool fibres, and hereinafter for convenience, wool fibres will be referred to, but the process is applicable to all protein fibres and filaments including camel hair, cashmere, mohair, vicuna, guanaco, alpaca, silk, llama or blends of these with sheep's wool.

The resins which may be used in the process of the invention may be any resin which will impart a shrink-resist effect to wool fibres. For example it may be a copolymer of ethylene, vinyl acetate and methacrylyl chloride, such as that sold under the trade mark ZESET T (E.I. du Pont de Nemours); a pre-polymer containing free isocyanate groups which may be the reaction product of a polyhydroxy compound, an alkylene oxide and a polyfunctional isocyanate, such as that sold under the trade name SYNTHAPPRET LKF (Bayer); a polyamide/epoxide resin system, such as that sold under the trade names Polyamide 115 and Epoxy Resin GY 260 (CIBA(ARL)); a polyacrylate; a resin formed by the catalytic oxidation of an unsaturated fatty acid ester; a polybutadiene containing functional groups, for example a carboxylated polybutadiene (as described in Textile Research Journal, Vol. 36, pp. 724—729, 1966); or a thiol-containing resin.

A particularly useful class of resin which may be applied to wool fibres, e.g. tops or card slivers, to impart shrink-resistance are the condensates of epichlorohydrin and a polyamide. Such a condensate may be obtained by heating together a C₂ to C₁₀ saturated aliphatic dicarboxylic acid and a polyalkylene polyamine containing 2—8 alkylene groups and then reacting the polyamide with epichlorohydrin. These condensates usually include a quarternising agent, e.g. dimethyl sulphate,

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which is added during or after the condensation in order to stabilise the product. Resins of this type are described and claimed in British Patent No. 865,727 and are commercially available under the trade mark HERCOSETT (Hercules Powder Company).

We have found that these epichlorohydrin/polyamide condensation products are preferably applied to wool fibres which have previously been partially chlorinated e.g. with an acid/hypochlorite solution. When the resin-treated fibres are then treated with the soil-release agent an excellent shrink-resist and soil-release finish is obtained, as compared with that obtained from the use of the resin alone.

The soil-release agents employed in this invention are condensates of an alkylene oxide and terephthalic acid and a commercial example is the product sold under the trade mark CASSAPPRET SR (Cassella Farbwerke Mainkur). The alkylene oxide may be ethylene oxide, propylene oxide or a mixture of the two oxides.

The soil-release agent may be applied to the protein material at the same time as the resin is applied, for example from the same resin bath, provided that there is no harmful chemical interaction between the soil-release agent and the constituents of the resin bath. Alternatively the soil-release agent may be applied at some later stage in the processing of the wool fibres. For example, if the resin shrink-resistant treatment is applied to wool top or card sliver the soil-release agent may be applied at any stage thereafter, e.g. at the fabric or the garment stage. However, it has been found to be advantageous to apply the soil-release agent to the resin-treated wool top or card sliver before it is subjected to further processing. The soil-release agent appears to act as an antistatic and a lubricating agent for the resin-treated fibres so that fibre bonding is reduced, less fibre breakage occurs, the sliver drafts more easily and a better yarn is produced.

The soil-release agent may be applied in widely varying concentrations but concentrations in the range 0.5% to 20% of solids, preferably 1—5%, based on the weight of the wool fibre are normally satisfactory. It may be applied by any of the conventional means for applying reagents to textiles e.g. by immersion, by spraying or by padding.

The concentration of the resin applied to wool fibres can also vary within a wide range and depends to some extent on the nature of the resin and the degree of shrink-resistance and textile handle required. Generally a concentration of 0.5 to 25% of solids and preferably 1 to 10%, based on the weight of wool fibres is sufficient for most purposes.

The invention will be further described with reference to the following examples. Unless

otherwise stated, all percentage values are by weight.

In these examples the Kubelka-Munk function is mentioned and this is derived from the following equation:—

$$K/S = (1 - R)^2 / 2R$$

where K/S is the ratio of the absorbed to scattered light and R is the mean reflectance of the sample. The K/S ratio is a measure of the amount of soil on the fabric surface. The degree of soiling is the difference in the K/S values before and after soiling ($\Delta K/S$). The degree of soil release is the difference in K/S values after and before marking the previously soiled samples.

EXAMPLE 1

A knitted fabric was manufactured from wool fibres which had been treated with 2% of a condensation product of trimethylene diamine, epichlorohydrin and adipic acid. A portion of this fabric was then impregnated with a solution containing 50 g/l of an ethylene oxide adduct of terephthalic acid (Cassappret SR), squeezed to 100% pick-up and dried at 70°C.

The treated and untreated portions of the fabric were heavily soiled together by tumbling with $\frac{1}{4}$ " felt cubes ingrained with dirt until the fabric portions were uniformly grey. The percentage reflectance of each sample relative to magnesium oxide was then measured using the green tristimulus filter of a Zeiss Elrepho reflectance colorimeter. The Kubelka-Munk function of these reflectance values was used as a measure of the apparent soil on the samples.

Each fabric portion was washed for 20 min. in 2 g/l soap at 40°C., and the improvement in appearance as a result of washing recorded as a percentage reduction in Kubelka-Munk value. The portion treated with resin alone actually showed a 40% increase in apparent soiling after washing, presumably as the particles of soil disaggregated in the resin film, whereas the appearance of the fabric treated with the ethylene oxide adduct was improved by 30%.

Three further washing treatments were given to the fabric portions. After these, 73% soil had been removed from the fabric containing the soil release agent, but only 37% was removed from the fabric without it.

EXAMPLE 2

Two portions of fabric identical with those described in Example 1 were washed in soap as before, but in each case a dirty piece of wool fabric of equal weight was washed along with it.

Soil redeposited on the clean materials in the wash, recorded in terms of Kubelka-Munk values is listed below.

	AK/S
Wool treated with resin	0.35
Wool treated with resin and soil release agent	0.16

5

EXAMPLE 3

A quantity of dry combed 64s quality wool tops was continuously treated in a backwashing machine with 1.2% chlorine under acid conditions, subjected to dechlorination and neutralisation treatments, and finally impregnated with 1.85% of a polyamide sold under the name of HERCOSETT 57 (Hercules Powder Company) and with 5% of an ethylene oxide adduct of terephthalic acid (Cassappret-SR). For comparison two further quantities of wool tops were similarly treated except that for one the ethylene oxide adduct was omitted from the resin bath, and for the other the polyamide was omitted.

The three lots of tops were dried and then spun on the worsted system of 2/32. metric count yarn. The lot treated with resin alone gave some fibre breakage and nep formation during gilling but the other two processed satisfactorily. Fabrics constructed from these yarns by knitting to a plain structure were then washed together for 1 hour at 40°C in a horizontal drum type domestic washing machine with a severe mechanical action. Area shrinkage values after washing were as follows:

	Area Shrinkage (%)
Untreated	56
Treated with chlorine	36
Treated with chlorine and soil release agent	28
Treated with chlorine and resin	15
Treated with chlorine, resin and soil release agent	0

The above table shows that the soil release agent considerably improves the shrink-resist effect of the polyamide resin and this, together with its lubricating action during gilling, makes its presence of considerable advantage in the production of "shrink-resist" wool yarn.

WHAT WE CLAIM IS:—

1. Process for improving the soil-release properties of protein fibres or filaments which comprises applying to the fibres or filaments a resin capable of imparting shrink-resistance thereto and a soil-release agent comprising a condensate of terephthalic acid and an alkylene oxide.

2. Process according to Claim 1, wherein the resin capable of imparting shrink-resistance comprises a copolymer of ethylene, vinyl acetate and methacrylyl chloride; the reaction product of a polyhydroxy compound, an alkylene oxide and a polyfunctional isocyanate; a polyamide/epoxide resin; a polyacrylate; a resin formed by catalytic oxidation of an unsaturated fatty acid ester; a polybutadiene containing functional groups; or a thiol-containing resin.

3. Process according to Claim 1, wherein the resin is a condensate of epichlorin and a polyamide, said condensate being obtained by heating together a C₃ to C₁₀ saturated aliphatic dicarboxylic acid and a polyalkylene polyamine containing 2—8 alkylene groups and then reacting the polyamide with epichlorhydrin.

4. Process according to any preceding claim, wherein the resin is applied to keratinous fibres in an amount of 0.5 to 25% of solids based on the weight of the fibres.

5. A process according to any preceding claim, wherein the soil-release agent comprises a condensate of ethylene oxide and/or propylene oxide and terephthalic acid.

6. Process according to any preceding claim, wherein the soil-release agent is applied to the fibres in an amount of 0.5 to 20% of solids based on the weight of fibre.

7. Process for improving the soil-release properties of keratinous fibres as claimed in claim 1 substantially as hereinbefore described.

8. Keratinous or other protein fibres or filaments treated by the process of any one of Claims 1 to 7 or an article containing them.

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